Contract #22ST.23440-4-9054

CONTRACT REPORT NO. 1ST84-00071

Final report

Conical Fluidized Bed

Summary

A fully instrumented liquid/solid fluidized bed of conical design has been built. The conical design of the fluidization volume yields the expected increase in the ranges of sizes and densities of particles that can be fluidized. Further, particles of sufficiently different sizes and/or densities have been shown to stratify into separate beds or horizons as expected from theory. Smooth separation into several beds is not trivial and a novel design for the water inlet into the conical fluidization volume had to be developed before satisfactory stratification could be effected.

The measurement of the potential generated when the fluidized particles are charged is possible. If different fluidized beds possess different surface charges as well as different sizes and densities the potential of each bed can be measured separately by means of a set of two electrodes that can be moved vertically in the conical vessel. The effects of acids, bases and any other surface active

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chemicals can thus be studied in situ within the volume of each of the different fluidized beds.

Other instrumentation includes a pH monitor as well as conductivity, temperature and pressure sensors to further study the behaviours of the liquid, particles and overall fluidized bed.

The data from all the instruments is collected and stored in a microcomputer and is thus available for further manipulation. Data acquisition is relatively slow in this system and therefore real time display and manipulation of the incoming data is feaseable.

The overall performance of the system as available, prove it to be ready for calibration and testing on samples of coal and clays to determine the effectivness of various factors and/or chemicals in altering the surface charges of particles in a water suspension and hence the effectivness of these factors in promoting flotation or flocculation.

Knowledge of these factors will lead to a more systematic approach to the study of flotation and flocculation. This systematization should pay ample dividends in rationalizing, and hence improving practices in a area of technology where trial and error at the plant level, and its enormous attendant costs, has been the

operating principle because of lack of reliable laboratory data.

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Introduction

The system is now complete, including a serviceable data acquisition software package and finished data acquisition hardware. Major additions since the last report have been the installation of the various probes, associated instrumentation and the data acquisition system. This report is structured as the previous report dividing the discussion of the new work into logically separate units. The summary covers the contract in its entirety.

Electrical and pressure probes

The electrical probes which measure the fluidization potential and the pressure probes which report the fluidized beds densities are both carried in the same assembly which can sample at different heights within the cone. The electronic components and the probes are mounted on a vertically movable platform. The vertical movement is achieved by using a Unislide™ assembly equipped with a vernier scale for position accuracy and with a resolution of 1 mm. per screw turn. See photo 1, 2 and 3 for assembly details. The platform supports the pressure transducer and a very high impedance imput amplifier ($<10^{12}$ ohms) which was locally designed and made. The very high capacitance is necessary since streaming potentials have a very high source

impedance (i.e relatively few ions move in the solution) and can be easily loaded down by the input impedeance of standard commercially available devices. Several design were tried to obtain optimum performance. The supplied device has an amplification of 10 to 1 as well as the desired input inpedance; the amplifier is also powered from its own independent power supply to avoid noise due to cross-talk, antenna pick-up and ground loops to other electronic devices. This power supply is mounted on the vertical alluminum "I" beam which also supports the Unislide™ assembly and the counterweight necessary for the smooth motion of the platform connected to the Unislide™.

The pressure sensor is a Valydine™ DP8 unit with a full range sensitivity of 25 mm of water (approx. 2mm Hg) and an ultimate resolution of about .013 mm of water when used with the associated Baker™ instruments electronic control unit.

Both the high input inpedance amplifier and the pressure sensor (but not the Baker instrument control unit) are mounted above the movable platform. Also on this platform are, a two way gas valve used to zero the differential pressure between the two inlets of the pressure transducer and a three way gas valve which allows gas to be admitted to the pressure sensor assembly. Great care should

be taken that the two way valve be open before gas is admitted to the system. Failure to do so will easily result in a ruptured pressure membrane in the DP8 unit.

The assemblies which transmit both the electrical and pressure information to the appropriate transducers hang under the movable platform and feed through the top plate of the cone assembly into the fluidized bed system. Each of these two assemblies consist of a thin walled glass tube of 3 mm. o.d., 2 mm. i.d., with a platinum wire running through it. The platinum wire is coiled at one end outside the tube and is connected, through a short length of teflon coated wire, to the high inpedance amplifier. The two assemblies are mounted so that their lower ends differ by 1 cm. in position. Thus the pressure and electrical potential information refects the differences across 1 cm of fluidized bed. The available accuracy of the instruments appears to be such that in future differences accross less than one cm. could be measured. The required electrode modification would be trivial. In the case of the electrical potential a common ground to a platinum screen situated at the cone inlet is also provided and hence information on the total potential across the fluidized bed to the upper assembly position is also available.

Feed through systems

Since the pressure and electronic systems operate outside of the fluidized bed cone and have to move with respect to it, the probes must connect two different enviroments, and not allow any exchange of gases, while allowing for relative movement. After some testing the best solution was found to be a flexible and compressible polyethylene sleeve mounted to the underside of the movable platform and connected to the top cover of the cone, see photo #2. This system works most satisfactorily under a slight overpressure hence a bubbler system has been provided to maintain this overpressure. This solution has the added advantage that should a leak develop no outside air will leak into the fluidized bed and contaminate the experiment.

Measurement of liquid characteristics

Probes to measure the main liquid characteristics, namely temperature, solution Ph and conductivity, were installed in the degassing box previously described. The electronic control and conditioning are provided by a Cole-Palmer solution analyzer capable of simultaneously outputting two of the three parameters as a D.C. level of +/200mv. full scale value.

Solid loading system

Testing showed that the loading of the solids to be measured before degassing of the liquid is unworkable. The degassing bubbles are generated mainly on the dispersed solid leading to flotation and transport of the solid through to the degassing chamber. Accordingly a loading system was designed which allows for the solid to be degassed while dry and concurrently with the solution, see photo #3. The system has been installed and tested satisfactorily.

Data acquisition

According to the previous discussion the following data channel must be acquired and displayed.

1) Pressure differential between the two assemblies, this value is position dependent and as such should be logged for every electrode position. After suitable manipulation these data will represent bed density.
2) The electrical potential difference between the two electrodes. This value is the measure of the fluidized bed potential developed between the two electrodes and is thus proportional to the surface (zeta) potential. This value is also position dependent and must be logged at every electrode position.

3) The electrical potential difference between the cone inlet and the uppermost electrode. This measures the total fluidized potential in the system up to the electrode position. It is position dependent and must be logged at every electrode position.

4) The solution temperature, presumably fixed, should only be measured sporadically to ensure that no temperature excursion has occurred.

5) The solution Ph is presumably fixed. The same considerations as 4) apply. Note however that much information about surfaces can be obtained by titration with acid or bases. If implementation of titration is desired continuous monitoring of Ph with time will is necessary and has already been implemented.

6) The solution conductivity. When dealing with well behaved surfaces, such as ballotini or quartz, this is a constant after an initial two to twenty hours conditioning period. Coal however is not a well behaved substance and previous experience strongly suggests the need for continuous monitoring.

Because of the characteristics of the Cole-Palmer analyzer,temperature and Ph are multiplexed on the same channel and hence only 5 channel sof data are necessary. If

the highly desirable automatic method to raise or lower the electrodes is installed six channels will again become necessary, the sixth being electrode position.

Given the amount of data to be colected an on line computer was deemed necessary. The system of choice consists of:

a) An apple IIe™ with 64k of memory, one disk drive, Monitor III™ display, Grappler™ printer interface and an Epson™ RX80 printer

b) An ADA-LAB™ card from Mandel scientific which resides in the Apple. This card offers a 12 bit double slope integrating A/D converter which is capable of twenty conversions per second as well as a 1.23 MHz. counter, a 12 bit 50 KHz. D/A and 8 bits of digital I/O.

c) An ADA-AMP™ external amplifier with options 1 and 2A also from Mandel scientific. This amplifier connects to the ADA-LAB card and provides computer controlled amplification (option 1) as well as 8 channels of double ended (i.e. differential) multiplexed input to the A/D (option 2A).

d) The QUICK-IO™ software which is an integral part of the ADA-LAB system.

Testing

Testing of the completely assembled conical fluidized bed system revealed the expected minor flaws and operational eccentricities.

In particular, a way to efficiently purge air below the inlet assembly and a finer control of flow than possible with the available direct pump control were necessary to obtain a proper bed stratification. A simple line feeding water and purged air to the degassing unit from the inlet unit, thus providing a bypass to the cone, solved both problems. Very fine flow control is now available by suitably choking this bypass line.

Operationally great care must be taken to avoid flooding the pressure sensor with water during the evacuation/nitrogen purge cycle necessary to remove carbon dioxide efficiently. Keeping the zeroing and gas purging valves on the gas sensor constantly open during these operations appears to be satisfactory.

As noted in the first report the verticality of the cone axis is critical to fluidized bed stratification.

Several interesting, and to my knowledge not previously reported, characteristics of conical beds were observed during this period. Since this observation proved to be easily reproducible a videotape of these phenomena is included in the report to the scientific authority and

further discussion will refer to these observations as well as to diagrams and figures included in the appendix.

The voice-over in the video tape will explain the main features. Only two points reguarding the video presentation need be discussed in this report.

First the bed entrainment and bed mixing behaviour is a function of loading and individual bed characteristics. This can be easily perceived by comparing the video with the voice-over and the video without voice-over. The very heavy entrainment which is visible with the lesser loading and smaller beds (no voice-over) is in fact very difficult to obtain with the higher loading. This seems to be connected with the formation of essentialy fully packed beds at the top of each individual fluidized bed.

Second, a certain amount of cross talk seems to be taking place in the pressure sensors. Because of the very high impedance amplifier the discharging of the electrode is slow leading to slow settling of the pressure sensor which is related to changes in electrode potential and not to actual pressure effects. This phenomena is well in hand and correction is possible both at the hardware and software level.

Reference to figures (1 & 2) in the appendix will clearly demonstrate further features of this system not seen

in the video report. These figures represent an elaboration of data obtained during a titration run. The electrode's position and the flow volume were not altered during the run and hence all the effects seen are due to changes in surface charge on the ballotini.

The electrodes were just immersed in the blue bed but did not enter the white ballotini bed. Thus the difference electrodes measure only the dependence of the blue ballotini surface charge on pH while the total represent an integration of all the fluidized beds present.

Three distinct set of values are charted representing the charges as seen by the difference electrodes, displayed on the extreme right, the total electrodes, displayed as a dark fill-in and the pH electrode seen as a white trace in the dark field of the total electrode.

The following features are noteworthy:

a) Addition of acid results in an overshoot of the pH value which takes about 3 minutes to re-equilibrate.

b) The titration curve follows a typical strong acid strong base pattern with pK of about 7. The curve is however stretched out by the presence of a reactive surface.

c) The two surfaces are different from each other as seen by the fact that the total potential goes through a maxima as the pH changes most rapidly. The difference

electrode reaches a plateau later. This effect is highlighted on figure 2. The dark band in this figure is proportional to the difference between the difference and total electrodes. A minimum is clearly visible. Further at high pH ,i.e at the top of the chart, the total electrodes are very sensitive to acid addition leading to overreaction and compensation while at lower pH the difference electrodes appear more sensitive to acid addition. A vertical scan at different electrodes positions should thus clearly be able to differentiate between the different beds. The difference data is very sensitive to bed variation between the two electrodes.

d) Above the pK point a very strong buffering action is exerted by the solids in the fluidized bed as evidenced by the minimal change in pH with addition of acid.

Further titration showed a gradual change in potential with no change in pH thus clearly indicating reaction at the solid surfaces. The behaviour indicated that the blue ballotini were responsible. Figure 3 illustrates this. Since the pH is an essentially straight line it was omitted for clarity. Figure 3 is easily interpreted when realizing that the total electrodes is also sensitive to the blue beads. Figure 4 shows the total run representing the

addition of 35 ml of 1 M HCl to the system initially at pH 10.4. Again the pH is omitted for clarity. The two potentials act in obviously different ways. To obtain a full data run the pH should have been dropped to about 2.5. The data displayed in figure (4) represents 2048 points taken at 10 seconds intervals, that is about five and one half hours. Further the 10 minutes equilibration time allowed during this tryal run was barely enough. Therefore an accurate run over the full pH range is estimated to need 8 hours, full automation of the titration experiment is thus imperative.

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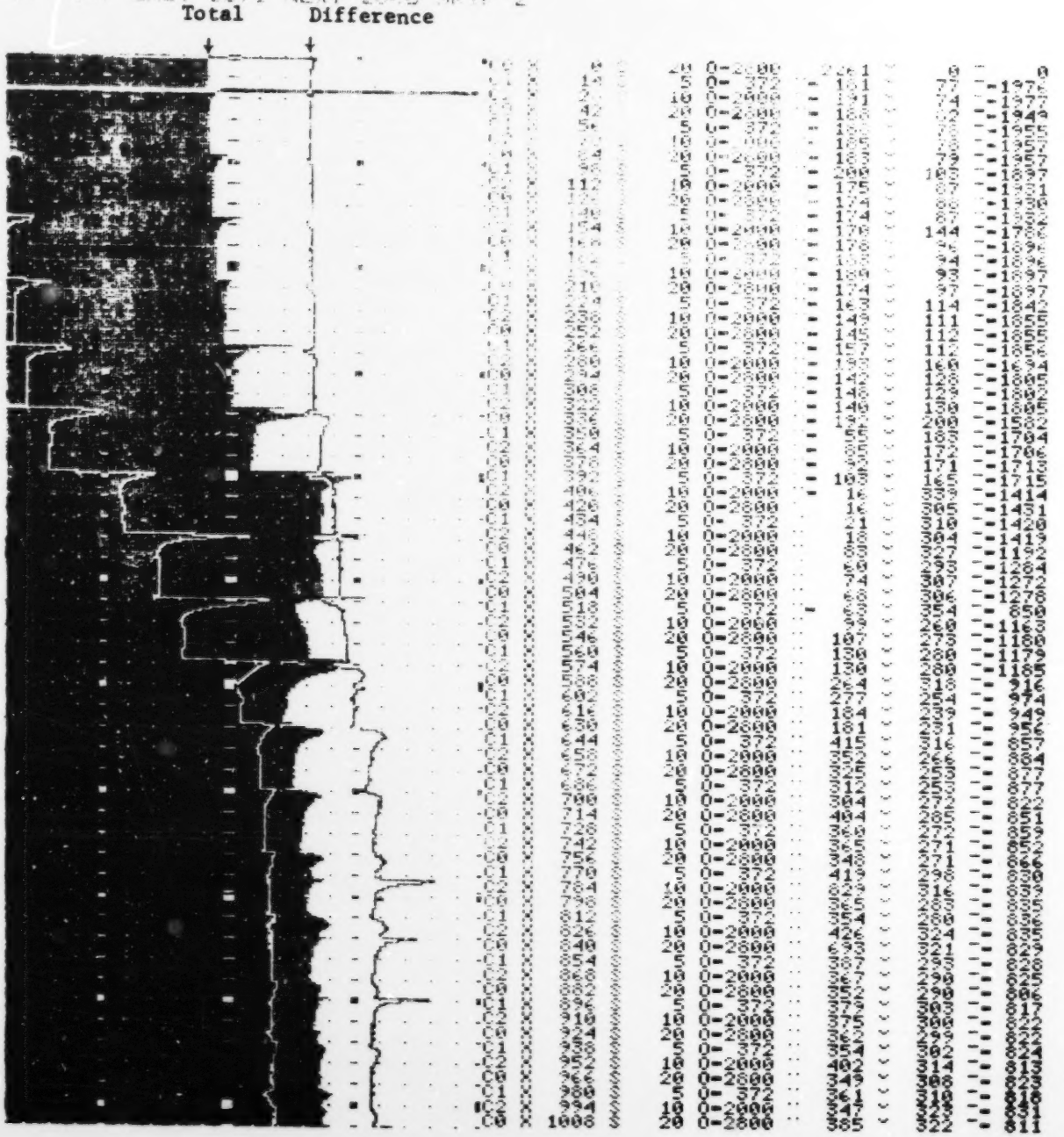
Figure 1



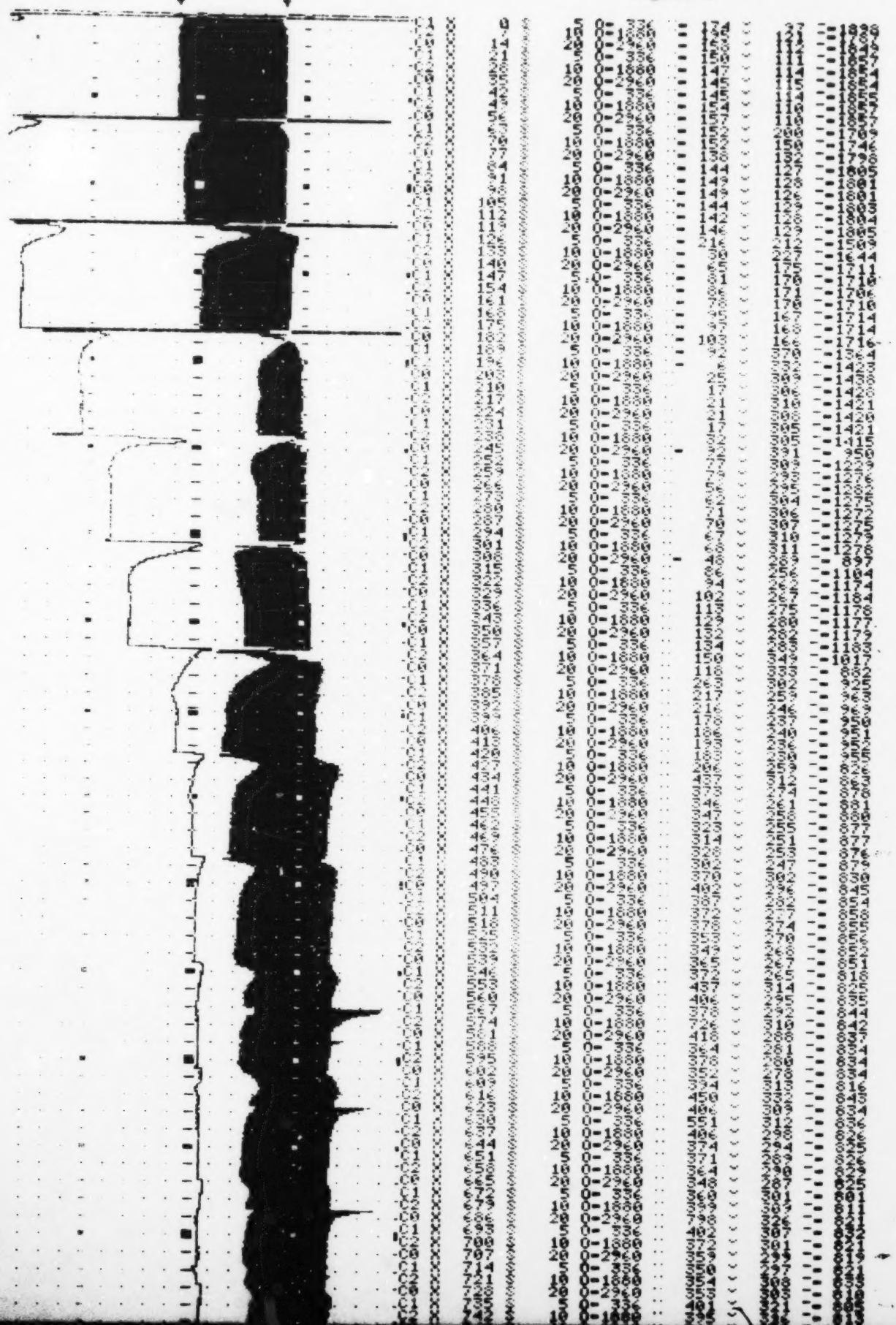


Figure 2

Figure 3

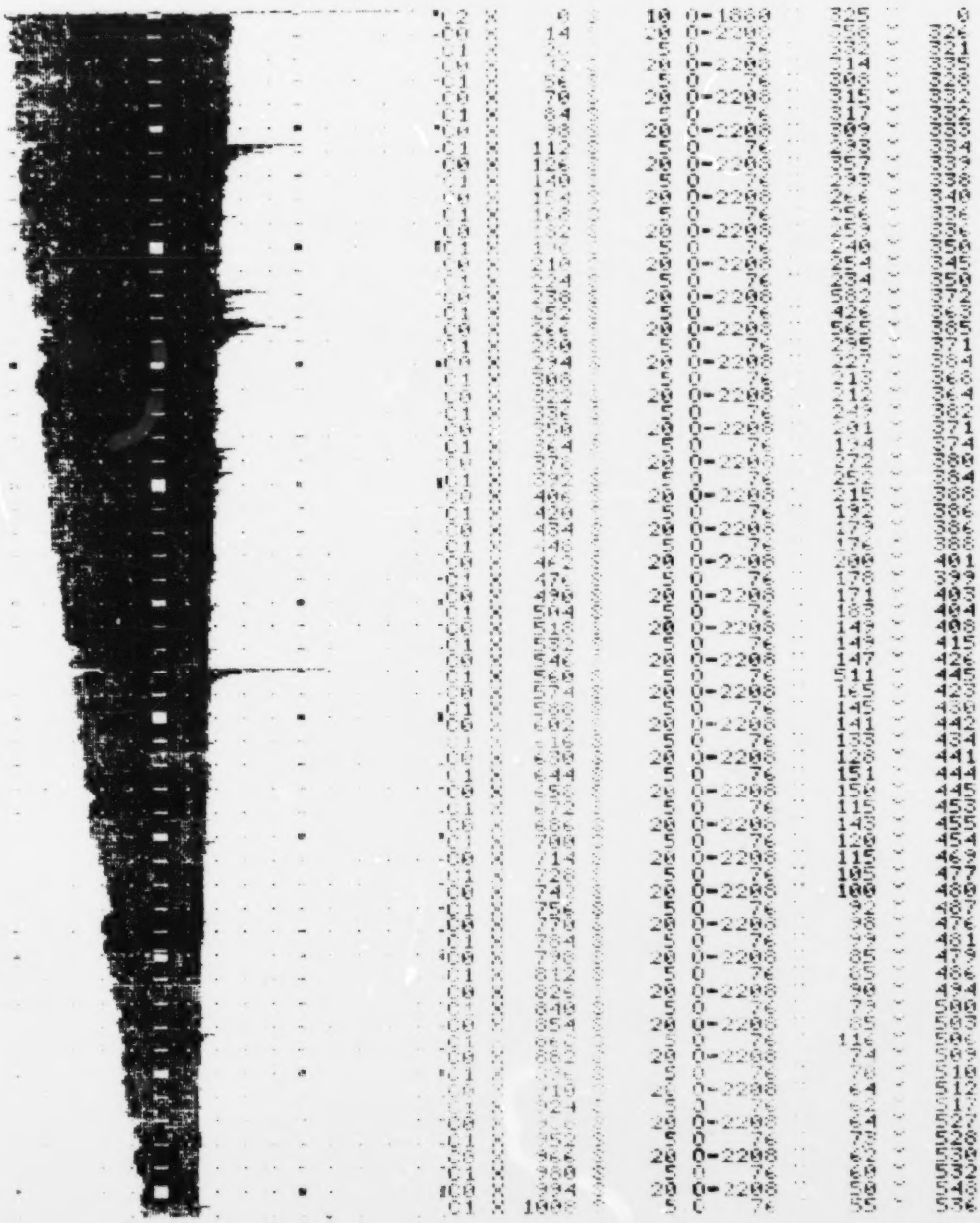


Figure 4

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?OUNT 0   WIDTH 200 TELESC 1  DLAY1
?ARG 0    RMARG 279 OSXCON 15 SPACE 1

?ANNL 0   STATUS 2  PRTROW 64 DECPNT 0
?CL  1    YSCL 10   OFST-1128 DOT-1
?ST 0     LAST 2047 NEXT 0    SKIP 4

?ANNL 2   STATUS 2  PRTROW 64 DECPNT 0
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?ST 2048  LAST 4095 NEXT 2048 SKIP 4
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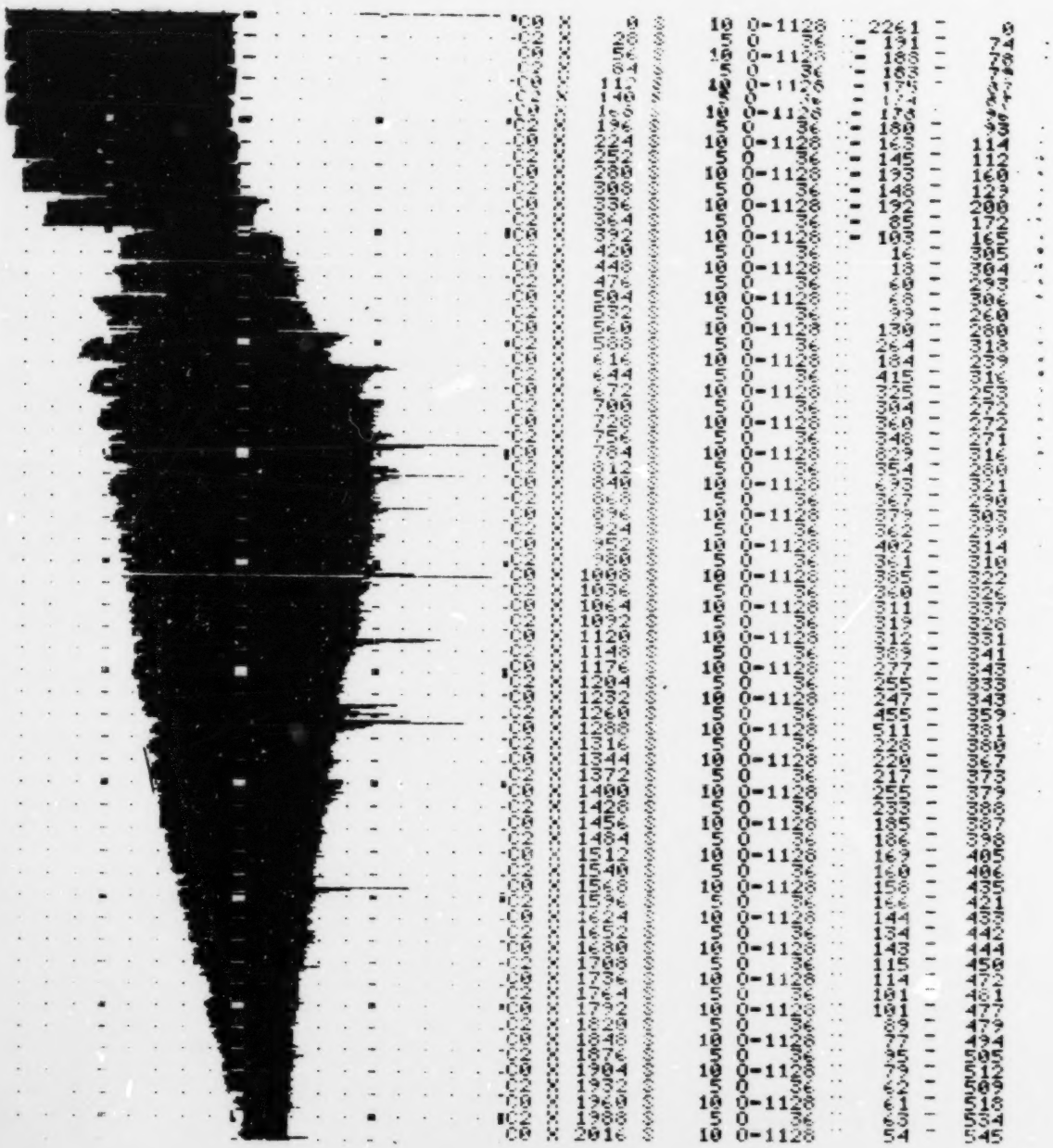

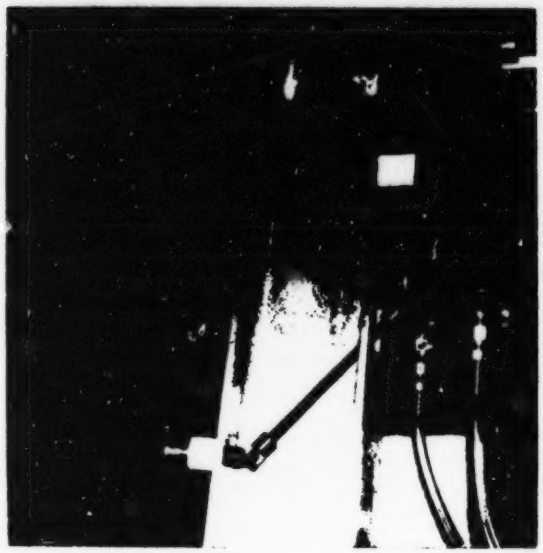

Photo 2

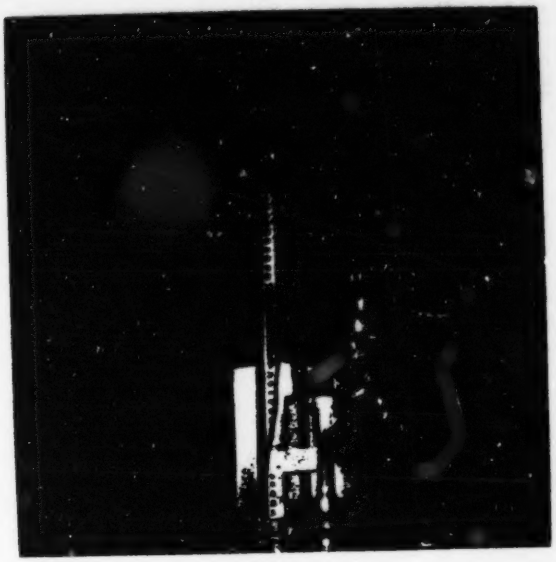

Photo 1

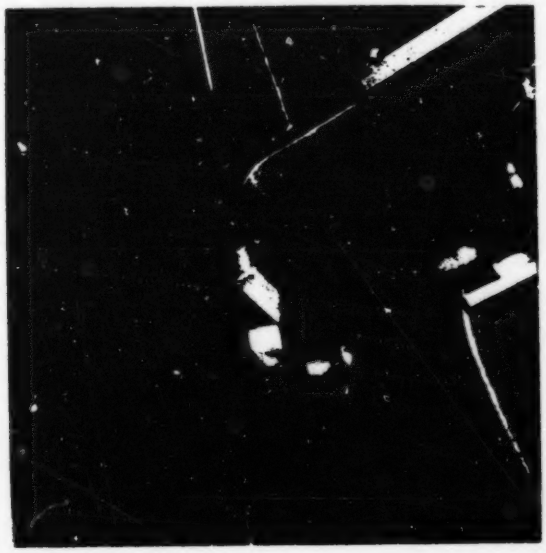

Photo 3

POOR

QUALITY

ORIGINAL